Submerged laser-induced plasma amplification of shockwaves using shock tubes for nanoparticle removal

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Abstract—Amplifying the dynamic pressure of submerged laser-induced plasma (LIP) shockwaves using shock tubes is introduced and demonstrated. The higher the amplitude of the pressure generated, the smaller the particles that can be removed, thus proving more useful for a variety of nanoparticle removal applications. Limiting the expansion of the submerged LIP core with a shock tube is a non-contact approach to increase pressure amplitude by nearly an order of magnitude for removal of particles, thus minimizing both shockwave and LIP radiation heating damage on the substrate. Radiation heating is identified as one of the major causes of damage in LIP nanoparticle removal and increases as the distance from the LIP core to the substrate decreases. However, submerged LIP shockwaves away from the core demonstrate a pressure decrease in the order of 70% every 5 mm and afterwards remaining comparatively steady. This demonstrates that the shock-tube technique results in higher pressures at distances significantly farther from the core of submerged LIP. In the current investigation, the effect of a set of shock tubes to amplify the transient pressure of the LIP-generated shockwave fronts has been studied to evaluate their specific pressure amplification performances. The effectiveness of a shock tube is quantified in terms of its pressure amplification factor. Through experimental data from several shock tube geometries examined, pressure amplification factors of 8.95 have been experimentally verified which is a ratio of shock tube generated submerged LIP transient pressure of 6.48 MPa to a transient pressure of 724 kPa without a shock tube at the same gap distance \( d = 2.5 \) mm. The potential advantages of shock tubes as an underwater amplification approach for predicted 10 nm particle removal are discussed.

Keywords: Submerged laser-induced plasma cleaning; pressure amplification factor; sub-10-nm particle removal; nanoparticle removal; shock tube; underwater LIP.

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1. INTRODUCTION

Generating strong pressure fields with the expansion of a submerged laser-induced plasma (LIP) core in a non-contact manner (i.e., without contact to a solid) on a substrate can be used for various practical applications, such as selective precision removal of nanoparticles from substrates including patterned wafers and extreme ultraviolet lithography photomasks. In the semiconductor industry, feature sizes are shrinking to a nanometer scale because of the demand for faster and more efficient integrated circuits; as a result, the tolerable particle size on the substrates is currently being reduced to sub-100-nm levels. According to the 2006 update of the International Technology Roadmap for Semiconductors (ITRS) [1] for front-end processes the projected future specifications are as follows: the removal of 90-nm particles by 2008, 65-nm particles by 2010 and 45-nm particles by 2013. ITRS 2006 also predicts that Extreme Ultraviolet Lithography (EUVL) masks will necessitate that 46-nm-sized defects be removed by 2008, 36-nm-sized defects by 2010 and 26-nm-sized defects by 2013. Following the guideline of the ITRS, the requirement for removal of sub-100-nm particles without substrate damage is a major issue in semiconductor manufacturing and will continue to guide the industry in coming years. However, keeping feature structures intact and adhering to an increasingly more stringent defect-density requirement will ultimately determine the effectiveness of a particular removal process.

2. LASER-INDUCED PLASMA TECHNIQUE

With LIP-generated shockwaves the effectiveness of generating removal forces sufficient to detach sub-100-nm polystyrene latex (PSL) particles from silicon substrates has been demonstrated in air [2, 3] and has directed further study of this technique and the utilization of shock tubes for smaller particles. In the current submerged LIP experiments, a pulsed-laser beam is focused underwater and plasma is initiated near the focal point of the lens due to the high intensity electric field as depicted in Fig. 1a. The plasma generated underwater, caused by high temperature and pressure, assumes the form of a three-dimensional near-ellipsoid and from its boundary a shockwave with supersonic velocity is created that departs outward normal to the boundary indicating a conversion of optical energy into mechanical energy [4, 5]. This vaporization of water forms fast expanding cavitation bubbles that can be used to remove particles [6, 7]. In the current LIP particle cleaning/removal procedure, the shockwave front is directed onto the particle to break the nanoparticle–substrate adhesion bond [8]. The two main process parameters in the submerged shock wave LIP approach are listed as (i) gap distance ($d$) defined as the distance between the center of the plasma core to the surface of the transducer (or substrate) (Fig 1b) and (ii) exit gap distance ($d_g$) defined as the distance from the exit of the shock tube to the transducer (or substrate). These parameters drive the effectiveness of a specific shock tube and
Figure 1. Instrumentation diagram of the experimental setup for submerged without shock tube LIP shockwave pressure measurement (a) and a schematic diagram of the submerged shock tube pressure amplification setup (b). The gap distance ($d$) represents the distance from the LIP core to the transducer/substrate. The exit gap distance ($d_g$) represents the distance from the exit of the shock tube to the transducer/substrate.
provide process design parameters for the LIP cleaning process. Direct contact of the plasma core \((d \leq 1 \text{ mm})\) with the substrate may lead to damage and must be avoided. In LIP nanoparticle removal without direct LIP contact with the substrate surface it has been recently determined that the two major sources of damage from LIP are both thermo-mechanical effects, i.e., stress fields created by the thermal expansion of the substrate due to the heat transfer from the hot gas behind the shockwave front, and radiation heating effects \([9, 10]\). Based on this observation, it is concluded that minimizing the effect of both the thermal field and radiation is a critical step in damage mitigation for sub-10-nm particle removal. The strength of the thermal field generated by the LIP core is proportional to the pulse energy of the laser and the gap distance \((d)\); therefore, a method that decreases the laser pulse energy and/or increases the gap distance while increasing shockwave front pressure is of practical interest, and finding the optimal gap distance for a given particle size range at which there is maximum removal without substrate damage is of practical importance. As the gap distance \(d\) (from the submerged LIP core to the substrate) decreases to the threshold distance, the LIP can damage the substrate due to heat transfer from the plasma core and the shockwaves which can induce considerable thermo-elastic stresses in the substrate \([11]\). This observation implies that the gap should be near the critical gap distance at which the thermal field is unlikely to damage the substrate to allow for maximum removal of the smallest size particles.

3. PRESSURE AMPLIFICATION

In the current study, the effect of a set of shock tubes to amplify the transient pressure of submerged LIP-generated shockwave fronts has been investigated to evaluate each tube for its pressure amplification ability and peak transient pressure performance in a liquid medium. A shock tube is essentially a pipe with one end blocked and a narrow aperture on the side in which a focused laser beam enters, in order to form LIP on the inside of the shock tube cavity (Fig. 1b). The principle motivation behind shock tube use for pressure amplification is based on the cavity of the tube creating a geometric constraint. This constraint essentially limits the expansion of the LIP core and increases its subsequent pressure shockwave front, consequently resulting in a stronger pressure field produced at the exit of the shock tube. Both submerged and in-air shock tubes deliver higher pressures at distances significantly farther from the point of LIP when compared to their LIP counterparts without shock tubes \([2, 3]\). Higher pressures generated from shock tubes are quantified in terms of the pressure amplification factor \((\Phi)\) defined as the ratio of the peak submerged shock tube generated transient pressure to the submerged shock tubeless LIP transient pressure while evaluating at an equivalent gap distance \((d)\).

The objective of the current study was to characterize and evaluate the efficiency of six shock tube designs by measuring their effectiveness in maximizing the pressure amplification factor \((\Phi)\) obtained from transient pressure measurements for a given Q-switched pulsed-laser. Six shock tubes with different dimensions (Fig. 2)
Figure 2. Schematic diagrams of the six cylindrical shock tube designs (ST1–ST6) used in the experimental study. All dimensions are in mm.

were evaluated based on the premise that restricting the volume for expansion of the shockwave will increase the pressure.

4. EXPERIMENTAL

The shock tubes used in this experimental investigation were cylindrical tubes made of brass with an aperture of 1 mm in diameter located along the length of the tube, while having one end of the tube sealed. The major design parameters considered for designing shock tubes were length, cavity depth and inner wall diameter. Six shock tubes (denoted ST1–ST6) were designed and their dimensions were determined from consecutive iterations on each previous design [3] (Fig. 2).

A single laser pulse is directed towards a 45° mirror with a wavelength specific anti-reflective coating, and then passes through a plano-convex lens which focuses the beam while entering the water to a point underwater and inside the cavity of a submerged shock tube to generate LIP (Fig. 1b). Due to the geometric constraint
of the cavity of the shock tube, the consequent pressure front is directed out at the exit of the shock tube towards the pressure transducer positioned at a distance \(d_g\), termed as the exit gap distance (Fig. 1b). This exit gap distance provides a process design parameter between shock tubes for optimizing the LIP cleaning process. The experimental setup (Fig. 1a) consisted of a Q-switched Nd:YAG pulsed-laser with wavelength of 1064 nm, a pulse energy of 370 mJ, a pulse width of 5 ns, a repetition frequency of 10 Hz and a beam diameter of 5 mm. A 40-mm-diameter mirror with a 1064 nm specific anti-reflective coating set at a 45° angle was used to steer the beam to the lens. The 100-mm focal length plano-convex lens was utilized to begin convergence of the beam in the air, then passing into the water to focus at a spot 40 mm below the surface of the water. This routine was repeated for both without (Fig. 1a) and with (Fig. 1b) the shock tube. A dynamic pressure transducer (Kistler, 603B1) with a central resonance frequency of 500 kHz (a rise time of 1 µs) was employed for pressure measurements. This transducer measures the average transient pressure across its active surface and was selected mainly for its bandwidth based on a set of waveforms acquired from previous work [12]. It is reasonable to anticipate that the point-wise transient pressure could be higher than the average pressure measured by the transducer. The charge output of the transducer was converted into voltage output using a dual-mode charge amplifier (Kistler, 5010B). The output from the charge amplifier was connected to a digitizing oscilloscope (Tektronix, TDS 3052) generating transient pressure–time waveforms which were acquired and stored in a computer for signal processing and further analysis.

In order to focus the laser beam into the aperture of the shock tube, the shock tube was mounted on a separate stage from the pressure transducer. The transducer was mounted onto a lateral 25-mm manual micrometer stage which allowed for varying the exit gap distance \(d_g\) in the experiments. This exit gap distance is the distance from the shock tube exit to the substrate surface and is useful when comparing effectiveness of different shock tubes. When \(d_g \approx 0\) mm, the cavity of the shock tube is nearly closed by the transducer and the pressures measured at these minimal gap distances are unusable due to the possibility that mechanical vibrations transmitted through the shock tube might result in damage to the substrate from contact with the shock tube as well as substrate contamination from the material of the shock tube.

The major design parameters considered for designing shock tubes were cavity depth and inner wall diameter (Fig. 2). Shock tubes ST1 and ST2 were utilized to study the effect of inner cavity diameter on LIP. ST3 had the same inner wall diameter as ST2; however, the cavity depth was shortened to 4 mm in order to study the effect of pressure and cavity depth. ST4 incorporated a diverging nozzle design with a slight increase in the depth of the cavity to 6 mm and a further decrease in inner wall diameter to 3 mm, which was used for investigating the pressure amplification due to the nozzle design. In ST5 the cavity depth was essentially lengthened to 11 mm, while keeping the inner wall diameter constant at 3 mm, allowing for a longer column for shockwave expansion, before emerging out of the
Submerged LIP shockwave amplification

Shock tube. ST6 incorporated a cavity wall diameter equal to that of the aperture at 1 mm and a 9 mm deep cavity inner diameter to study the effects of very small inner wall diameter and compare to the former shock tubes. All shock tubes investigated had an aperture to cavity base distance of 2 mm, while varying the aperture to cavity entrance length (based on total length of the tube) (Fig. 2). A summary of the six shock tube dimensions is available in Table 1.

Before the transient pressure measurement experiments were carried out, measurements without a shock tube were obtained with a gap distance ranging from \( d = 0.5 \) to 18.0 mm incremented in steps of 1.0 mm to acquire baseline data for comparison of the pressure amplification factor (\( \Phi \)). The optimal experimental water height was found by conducting a number of trials at a range of water levels. The optimal water level experimentation started by mounting a shock tube at a fixed height from the lens (94 mm). The water level was then determined from the central axis of the shock tube to the surface of the water. This distance was then incrementally increased in steps of 10 mm, starting from 10 mm ranging to 80 mm. Table 2

<table>
<thead>
<tr>
<th>Shock tube</th>
<th>( d ) (mm)</th>
<th>( P ) (kPa) at ( d_g = 0.5 ) mm</th>
<th>( \Phi )</th>
<th>Description</th>
<th>Inner wall diameter (mm)</th>
<th>Cavity depth (mm)</th>
<th>Nozzle</th>
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<tr>
<td>1</td>
<td>7.5</td>
<td>776</td>
<td>1.72</td>
<td>Long inner wall ( \varnothing )</td>
<td>5</td>
<td>9</td>
<td>No</td>
</tr>
<tr>
<td>2</td>
<td>7.5</td>
<td>3060</td>
<td>6.77</td>
<td>Medium inner wall ( \varnothing )</td>
<td>3.5</td>
<td>9</td>
<td>No</td>
</tr>
<tr>
<td>3</td>
<td>2.5</td>
<td>6480</td>
<td>8.95</td>
<td>Short cavity depth</td>
<td>3.5</td>
<td>4</td>
<td>No</td>
</tr>
<tr>
<td>4</td>
<td>4.5</td>
<td>2820</td>
<td>6.24</td>
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<td>6</td>
<td>Yes</td>
</tr>
<tr>
<td>5</td>
<td>9.5</td>
<td>1005.7</td>
<td>2.73</td>
<td>Long cavity depth</td>
<td>3</td>
<td>11</td>
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</tr>
<tr>
<td>6</td>
<td>7.5</td>
<td>3000</td>
<td>7.28</td>
<td>Small inner wall ( \varnothing )</td>
<td>1</td>
<td>9</td>
<td>No</td>
</tr>
</tbody>
</table>

The table also includes a description and side-by-side comparison of major attributes: inner wall diameter and cavity depth.

<table>
<thead>
<tr>
<th>Water height (mm)</th>
<th>30</th>
<th>40</th>
<th>50</th>
<th>60</th>
<th>70</th>
<th>80</th>
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<td>Max. pressure (kPa)</td>
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<td>4480</td>
<td>3960</td>
<td>4480</td>
<td>3760</td>
<td>3400</td>
</tr>
<tr>
<td>Lower range (kPa)</td>
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<td>3640</td>
<td>3560</td>
<td>3920</td>
<td>3040</td>
<td>2960</td>
</tr>
<tr>
<td>Range difference</td>
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<td>840</td>
<td>400</td>
<td>560</td>
<td>720</td>
<td>440</td>
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<td>Trial 1</td>
<td>1480</td>
<td>4440</td>
<td>3560</td>
<td>4480</td>
<td>3760</td>
<td>3040</td>
</tr>
<tr>
<td>Trial 2</td>
<td>1536</td>
<td>4420</td>
<td>3800</td>
<td>4280</td>
<td>3040</td>
<td>2960</td>
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<tr>
<td>Trial 3</td>
<td>1544</td>
<td>4480</td>
<td>3680</td>
<td>3920</td>
<td>3700</td>
<td>3080</td>
</tr>
<tr>
<td>Trial 4</td>
<td>1528</td>
<td>3640</td>
<td>3960</td>
<td>4080</td>
<td>3640</td>
<td>3400</td>
</tr>
<tr>
<td>Average pressure (kPa)</td>
<td>1522</td>
<td>4245</td>
<td>3750</td>
<td>4190</td>
<td>3535</td>
<td>3120</td>
</tr>
</tbody>
</table>
highlights the results from each of the water height steps. A water level of 40 mm generated the same maximum pressure as 60 mm level, however the average pressure at 40 mm level was 55 kPa greater. It was determined that the optimum water height was 40 mm. It should be noted that both 10 mm and 20 mm water levels resulted in significant splashing, which deposited water on the lens. If the laser is pulsed with water on the lens, the lens has a high chance of breakage. All experiments with the shock tubes (ST1–ST6) and their pressure waveforms were acquired at the 40 mm water level. It was noted in the shock tube experiments, these two design parameters for performance enhancement were of interest: gap distance \(d\) and exit gap distance \(d_g\). The shock tubes had different gap distances \(d\) measured from the LIP formation at the aperture to the substrate face. This distance varies accordingly to the dimensions of each specific shock tube (Fig. 2). The exit gap distance \(d_g\), however, was constant for all shock tubes and all experiments were carried out initially with a minimum exit gap distance of \(d_g = 0.5\) mm ranging to \(d_g = 11.0\) mm. For all shock tube designs the shockwave exits through the open end of the shock tube nearly as a plane wave. Experiments were conducted at ambient room conditions.

5. RESULTS AND DISCUSSION

In the experiments conducted, transient pressure waveforms underwater were obtained for both with and without a shock tube for comparison of the pressure amplification factors \(\Phi\). Waveforms were acquired for all six shock tubes ST1–ST6 (Fig. 2) for exit gap distance \(d_g\) steps of 1.0 mm. These waveforms were analyzed to determine the maximum pressure and the position of the shockwave front for each shock tube from the LIP core. Table 1 reports the transient pressures for each submerged shock tube at \(d_g = 0.5\) mm.

The peak submerged transient pressure amplitude \(P_{\text{max}}\) without a shock tube was measured as 1024.0 kPa at \(d = 0.5\) mm, with the maximum pressure decreasing with increase in the gap distance \(d\) (Fig. 3). The peak pressure is an average transient pressure measurement taken from across the active surface of the transducer face. Transient pressure measurements for the six submerged shock tubes were then characterized for their peak values for comparison against the case without a shock tube. The peak pressure obtained with ST1-H4 at an exit gap distance \(d_g = 0.5\) mm shifted from 776 kPa to 788 kPa at \(d_g = 1.0\) mm, and then to 576 kPa at \(d_g = 2.0\) mm (Fig. 4a). Except for ST1-H4, exit gap distance \(d_g = 0.5\) mm consistently gave the highest pressure and was used almost exclusively for peak pressure measurements. ST2-H4 generated the second highest pressure of 3000 kPa at \(d_g = 0.5\) mm (Fig. 4b). The maximum peak pressure amplitude obtained from all of the submerged shock tubes was for ST3, generating 6480 kPa at \(d_g = 0.5\) mm with a rise time of 1.25 \(\mu\)s (Fig. 4c). ST4 generated a maximum transient pressure of 2820 kPa at \(d_g = 0.5\) mm (Fig. 4d), while ST5 achieved 1006 kPa at \(d_g = 0.5\) mm (Fig. 4e). Finally, the peak pressure attained with ST6
Figure 3. Peak transient pressure measurements $P_{\text{max}}$ for submerged LIP shockwave generation for varying gap distances $d$.

Figure 4. (a) Transient peak submerged pressure measurements $P_{\text{max}}$ for shock tube 1, hole 4 (ST1-H4) directly compared against the water only case. The gap distance ($d$) ranges from 7.5 mm to 18 mm. Note that the pressure is always greater than the water only case.

reached 3000 kPa at $d_g = 0.5$ mm (Fig. 4f). The peak pressure amplitudes obtained with each of the six shock tubes and also without the shock tube, while evaluating the transient pressure waveforms for pressure versus gap distance ($d$) are reported in Table 1 and Fig. 4. The pressure amplification factor ($\Phi$) versus gap distance ($d$) for all shock tube designs is presented in Fig. 5.

6. CONCLUSIONS AND REMARKS

The objective of the current investigation was to maximize the pressure amplitude created from the submerged LIP shockwave front for a given pulsed-laser using...
Figure 4. (Continued.) (b) Transient peak submerged pressure measurements $P_{\text{max}}$ for shock tube 2, hole 4 directly compared against the water only case. The gap distance ($d$) ranges from 7.5 mm to 18 mm. Note that ST2-H4 generated the second highest transient pressure at the furthest distance away, however at exit gap distances ($d_g$) of >5 mm pressure decreases rapidly to below the water baseline. (c) Transient peak submerged pressure measurements $P_{\text{max}}$ for ST3 directly compared against the water only case. The gap distance ($d$) ranges from 2.5 mm to 13 mm. This shock tube generated the highest transient pressure, however at a closer distance compared to the other shock tubes. ST3 also exhibits a much slower decay of pressure as the substrate is moved further from the LIP.

Shock tubes in order to increase the gap distance and thereby decrease the possible thermo-mechanical damage per unit pulse energy of the laser. Shock tubes can also decrease the LIP energy required for generating a higher particular pressure level at the same gap distance or the same pressure at a farther gap distance ($d$). This approach may lead to the use of smaller pulsed-lasers for desired nanoparticle cleaning applications. The main aspects of the current investigation were varying the shock tube parameters and to test the effects of inner wall diameter, cavity length and water depth on the transient pressure generated. The effects of these shock tube
design parameters in amplifying the transient pressure amplitude were successfully evaluated with respect to their differing geometric constraints associated with the submerged LIP propagation and expansion. The resulting pressure amplification factors are compared to understand the effect of geometry of the shock tube on the shockwave pressure generated at the shock tube exit. It is determined that shock tubes substantially intensify submerged shockwave pressure and, as a result, can remove smaller particles with stronger adhesion bonds.

Shock tube 3 (ST3) recorded a peak transient pressure of 6.48 MPa measured at a gap distance \(d\) of 2.5 mm, resulting in a peak pressure amplification factor \(\Phi = 8.95\). ST6 recorded a peak transient pressure of 3000 kPa measured at a gap distance of 7.5 mm, resulting in a \(\Phi = 7.28\). ST6 generated less transient

Figure 4. (Continued.) (d) Transient peak submerged pressure measurements \(P_{\text{max}}\) for ST4 directly compared against the water only case. The gap distance \(d\) ranges from 7 mm to 17 mm. (e) Transient peak submerged pressure measurements \(P_{\text{max}}\) for ST5 directly compared against the water only case. The gap distance \(d\) ranges from 9.5 mm to 15 mm.
Figure 4. (Continued.) (f) Transient peak submerged pressure measurements $P_{\text{max}}$ for ST6 directly compared against the water only case. The gap distance ($d$) ranges from 7.5 mm to 18 mm.

Figure 5. The pressure amplification factor $\Phi$ as a function of the gap distance $d$ for each shock tube design. The initial points (leftmost) are for exit gap distance of $d_g = 0.5$ mm.

pressure than ST3, but did so at a much further gap distance. In applications where possible damage from thermo-mechanical effects is a concern, the distance from the LIP core to the substrate/transducer is of utmost importance, making ST3 a viable option. Greater pressure could possibly even be arrived at by situating the laser directly above the converging lens, thus eliminating the need for a 45° reflecting mirror. This arrangement would prevent energy losses due to mirror reflections.

The submerged shock tube amplification method could be beneficial for various applications such as damage-free sub-10-nm-sized particle removal from substrates which may include patterned silicon wafers and extreme ultraviolet lithography photomasks. The peak pressures and pressure amplification factors together
demonstrate that a trend toward optimization for even greater transient pressures is possible, proving shockwave amplification of LIP by shock tubes to be useful.

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